Erosive Wear Mechanism of New SiC/SiC Composites by Solid Particles

EROSIVE WEAR MECHANISM OF NEW SiC/SiC COMPOSITES BY SOLID PARTICLES

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ABSTRACT

Solid-particle erosive wear test by impinging SiC powders was carried out at room temperature over a range of median particle sizes of 425–600 μm, speed of 100 m/s and impact angle of 90° by wear measurements and scanning electron microscopy. Erosive wear behaviour was examined on newly fabricated NITE SiC/SiC composites and two commercial compositesby CVI and NITE fabrication route. Microstructural observation was performed to examine the correlation between erosive wear behaviours and fabrication impurities. Conspicuous defects were observed in the prototype materials as the forms of porosity, fibre deformation, residual oxide, PyC deformation, PyC cleavage, and, etc. Erosive wear behaviour was rather serious in prototype of fabricated composites, which employ Pre SiC-fibre and phenolic resin. Two dominant erosive wear mechanisms were observed, one is delamination of constituents mainly caused by erosive crack propagation. Another is

fragmentation and detachment of constituents, which usually resulted from erosive impact. A unit size of delamination was the most decisive factor on wear volume. Bonding strength of each constituent was mostly affected by various forms of porosities. Therefore, fundamental cause and consequent results will be carefully elucidated. In addition, to reduce the dominant wear by improving the fabrication conditions, the correlation of microstructural defect and wear behaviour was investigated. The final product of cost effective composite shows over 2.5times wear resistance in comparison to commercial CVI composite. Consequently, by controlling the fabrication impurities, the improvements have been successfully made for a new fabrication technique, as a result, the known defects are rarely observed in final product. Schematic wear model of erosive wear mechanismsare proposed for the newly fabricated SiC/SiC composites under particle erosion.

1. INTRODUCTION

Considerable efforts has been given in the evaluation of a SiC or erosion related ceramic and ceramic composite materials [1-42] under engineering components in such applications as gas turbine parts, sealing bearings, and burner parts [43-63] where all these applications may involve with solid-particle impact and erosive wear due to the necessity of a knowledge regarding behaviours and mechanisms before it can be used with confidence. However, most are focused on particulate composites and only a few works can be found for advanced fibre-reinforced SiC/SiC composites [41-42,61-63], and particularly some in impact and erosive wear [59-63]. Continuous SiC fibre-reinforced SiC matrix (SiCf/SiC) composites are a promising candidate for a structural application, particularly in severe conditions such as high temperature and wear resistant environments [59-73]. It is well known that SiC has one of the highest hardness of all single-phase ceramics, moreover, possesses fascinating performance in various conditions [61-73].Brittle behaviour is the dominant failure factor for most ceramics, but SiC_f/SiC composites have solved most of the inherent brittle issue. It is also known as one of the most advanced composite material systems due to the commercial availability as well as promising mechanical properties in elevated temperature. The use of SiC_f/SiC composites such as combustor liners and turbine vanes provides the potential of improving next-generation turbine engine performance, through lower emissions and higher cycle efficiency, relative to today's use of super alloy in high temperature components. The demand of the new advanced materials is increasing, especially in the recent high-efficiency advanced energy system and/or severe environments, at the same time the understanding of wear mechanisms can lead to improve material performance and also essential to design advanced tribosystems.

SiC_f/SiC composite is composed mainly by matrix, fibre, and matrix-fibre interface as a constituent part, which follows the definition of composite. A significant progress has been described in our previous works [65-72]. In case of the highly qualified grade SiC_f/SiC composite developed in Kyoto University, the Tyranno SA^{TM} grade fibre and the pyrolytic carbon (PyC) interface by CVD route are employed as fibre-reinforcement and fibre-interface, respectively. The mechanical properties are promising due to fascinating microstructures e.g. near-full dense, on-demanded distinct thickness of the interface, high crystallized grains, stoichiometric compositions, and no residual carbon or silicon area (see figure 1). State of the art in SiC/SiC composites has solved most of the potential issues of near-net shaping and machining, joining technology, and evaluation technology [73-90]. There are no doubts that SiC/SiC composites are at the forefront of advanced materials technology and prime candidates for structural material in severe environments. However, it still has issues of brittle reliability and extremely high cost due to the complication of many complex processes for production.

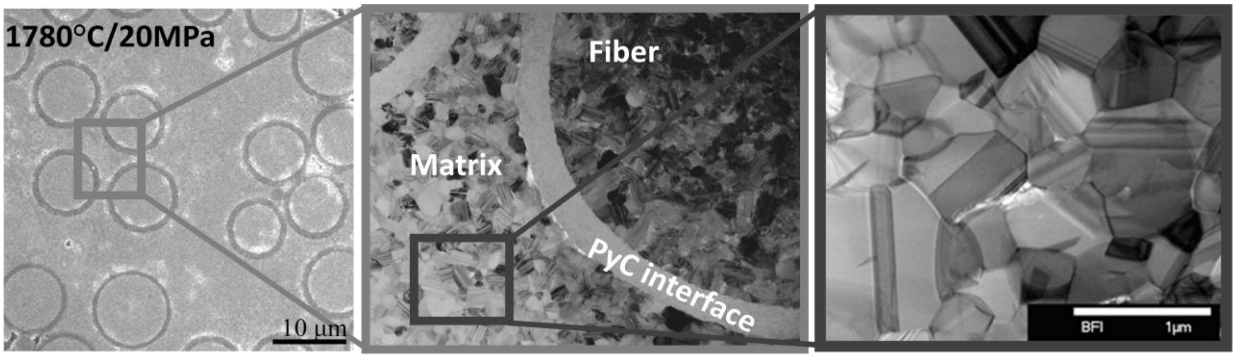

Figure 1.High densification and crystallization of high-qualified grade SiC_f/SiC composite.

A novel cost effective fabrication on SiC/SiC composites(the rest is omitted as a new SiC/SiC composite) has been developed for high erosion resistance. To accomplish both cost issues and foreign object damage (FOD) resistance, Pre-SiC fibre was employed as fibre-reinforcement with adopting a new phenolic resin as fibre-interface, in particular to substitute CVD coated PyC interface. In order to investigate the dominant wear mechanism correlated to crucial parameters of material fabrication to answer the purpose, a number of new SiC/SiC composites were fabricated.Wear control by improvement of materials and design of operating conditionshas become a strong need for the advanced and reliable technology in order to ensure the consequence of durability and reliability issues caused by wear.This study intended to improve not only erosion performance but material properties by following methods. Optimization of the fabrication conditions of prototype SiC/SiC composites through controlling the microstructural defects was accomplished by analysing the wear behaviours of all constituents of composites (matrix, fibre and interface).

2. EXPERIMENTAL PROCEDURES

2.1. Material preparation

Nano-powder Infiltration and Transient Eutectoid (NITE) process has been selected to fabricate SiC/SiC composites for superior characteristics [67-91]. It is a highly optimized liquid phase sintering

(LPS) process, available in commercial. Figure 2 shows the brief concept and procedures regarding manufacture of NITE process. Fabrics are prepared as a form of prepreg sheet after interface pre-coating by the methods explained in figure 3 for employing the PyC interface around the fibre tows. The prepreg is impregnated with the matrix precursor by passing through the slurry. A powder mixture of over 90wt% SiC and below 10wt% of sintering additives was prepared by milling process for raw material of slurry. To make a green body, the prepreg sheet is cut into a demanded size, stacked up in desired orientation, and warm pressed in a certain temperature. A burnout process was carried out to eliminate the organic impurities out of the sample specimen resulting in almost near-full dense composite. Finally, hot-pressed into a dimension of on-demanded size, in this study 40 mm x 20 mm sized plate was fabricated, under Ar atmosphere at 1850 °C, applying 20 MPa of load by a graphite mould. Figure 3 illustrates major differences offibre coating process in comparison of pre-existing method.Generally, the CVD coating produces on-demanded distinct and homogeneous thickness of coating layer, in opposite a large amount of time and processing complication is inevitable. According to the previous studies the porosity is one of a dominant factor on material performance against erosive wear [65-71]. The focus of fabrication was applying the easiest and cheapest coating method as the substitution of the CVD coating, nonetheless to fabricate distinct interface between the matrix and fibre. This new coating method using a phenolic resin needs the following procedures, infiltration of phenolic resin into the fibre tows, thickness control, and pyrolysis of phenolic resin. These processes anticipate givinga certain thickness of PyC around the fibre tows. Pre-SiC fibre is a state of polymer driven SiC fibre as a precursor of SiC fibre, that the mechanical strength is not decided yet, nevertheless it has a strong merit of commercial price competitiveness.

New SiC/SiC composites were fabricated in different conditions by modifying both the amounts of employed PyC and fibre heat treatment for interface-control and bonding-control among the fibre tows, respectively. Sintering conditions (e.g. temperature, applied pressure, and holding time) and fabrication compositions (e.g. amount of SiC powder and sintering additives) are remained as constant. Fabrication characteristics of the various composite materials used in this test are listed in table 1.

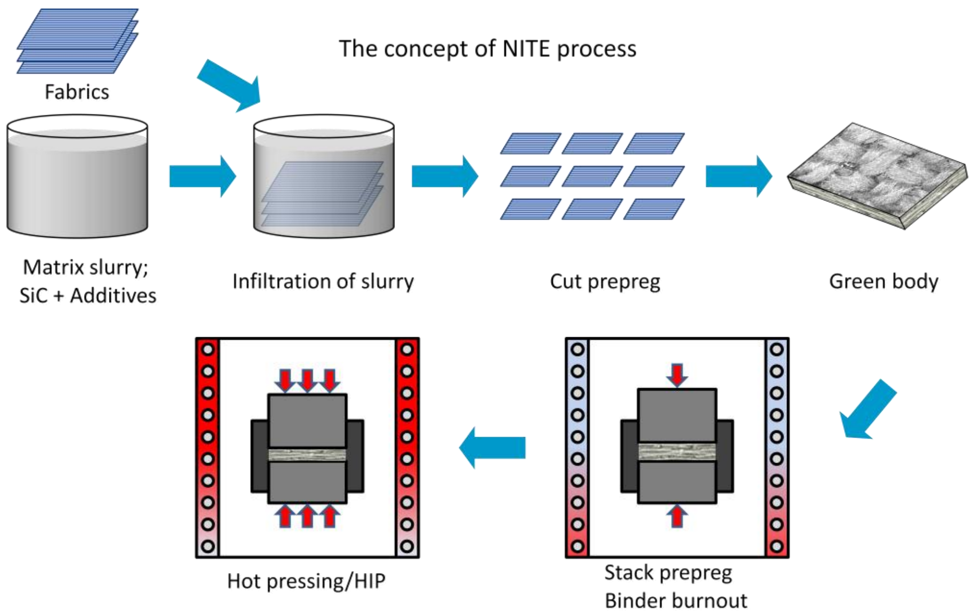

Figure 2.Schematic concept and process of NITE fabrication route.

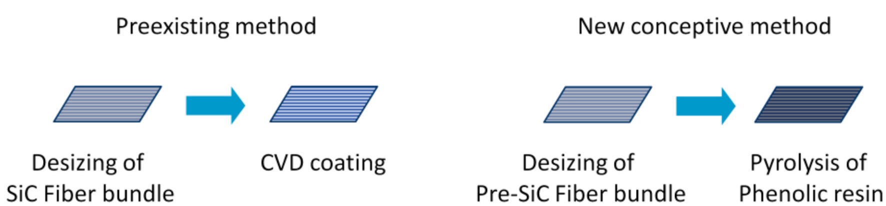

Figure 3.Schematic comparison of fabric preparation.

Table I. Fabrication characteristics of various SiC/SiC composites

SiC_f/SiC composites	Fabrication route	Employed coating	Fibre coating method	Fibre type	Thickness of employed PyC	Fibre heat treatment
Commercial product #1	NITE	PyC	CVD	Cef-NITE^{TM}	500 nm	N/A
Commercial product #2	CVI	PyC	CVD	Tyranno SA^{TM}	80 nm	N/A
Prototype(LOT#11)	NITE	PyC	Pre-pyrolysis	Pre-SiC fibre	Very thick	500 °C
Prototype(LOT#12)	NITE	PyC	Pre-pyrolysis	Pre-SiC fibre	Thin	500 °C
Final product(LOT#17)	NITE	PyC	Pre-pyrolysis	Pre-SiC fibre	Very thin	Delicately Controlled

2.2. Erosion wear test

Erosive wear test was carried out by impinging SiC powder (green carborundum #36) on the surface of a test specimen. Using jets to utilize repeated impact erosion with a small nozzle delivering a compressed gas containing those abrasive particleswhich coincident with ASTM G76, as depicted in figure 4. Figure 5 illustrates a close-up view of impact on the test specimen focused on surface wear, each of a broken line and solid line indicates the example of real surface before and after the test, respectively. When impinging the erodent on the composite materials, considering the difference of crack propagation rate on PyC interface and SiC matrix/fibre, the direction of reinforced fibres will play an important role for wear performance. The composite material shown in figure 5 is highly over exaggerated to emphasize the direction of reinforced fibretows; the erodent used in this study is over 42 times bigger than the fibre, for reference,average diameter of commercial SiC fibre is 7.5 – 14 µm.Table 2 shows the experimental conditions for the test. It is well known that the effect of impingement angle on wear rates is the greatest close to perpendicular for brittle materials.

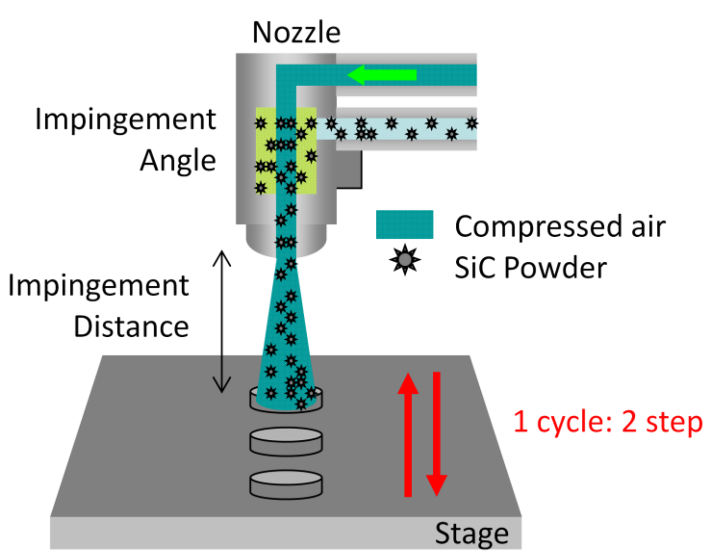

Figure 4.Schematic illustration of particle erosion environment.

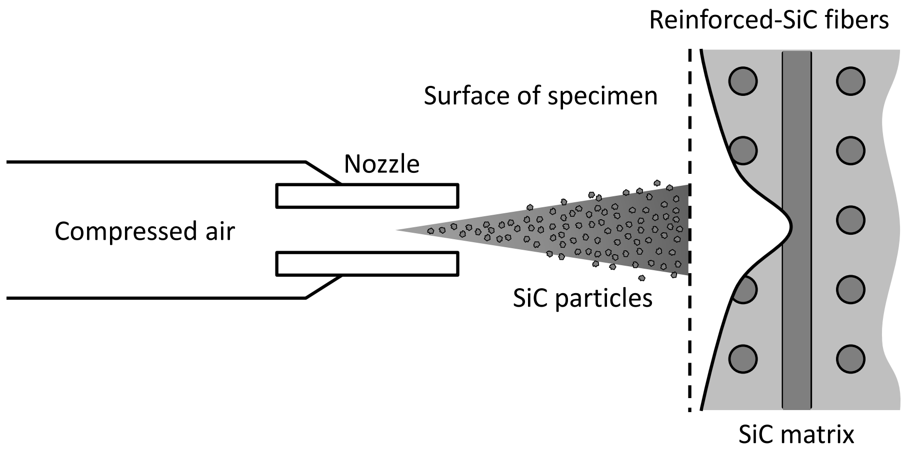

Figure 5 Schematic of the solid particle impingement using gas jet on the surface of a test specimen.

Table II Experimental conditions of particle erosion wear test

Test parameters	Erosion method	Erodent	Step of erosion	Impingement conditions		
				Angle	Distance	Jet pressure
Value	Solid particle compressed air	SiC powder [a]GC #36	3 steps	90 °	40 mm	5 atm

[a]Green carborundum (GC) is a bulk grinding compound generally for polishing purpose

Distribution of particle size is #36 is 600 ~ 425 μm

Average particle size is around 510 μm

A total of 100 g of particles are impinged on composites surface in 33 g increments were impacted at room temperature on the surfaces of the composite.Protection seal was adopted to protect the partial surface of a test specimen from severe particle impingement while the erosion tests. Detachment of the protection seal and removal of debris by air-gun precedes themeasuring of surface topography by a surface profilometer after each incrementof erosion. The average worn depth was calculated by the difference in topography between the baseline of uneroded surface and eroded surface. Erosive wear rate was calculated by the average volume of worn surface divided by the total area of eroded surface.

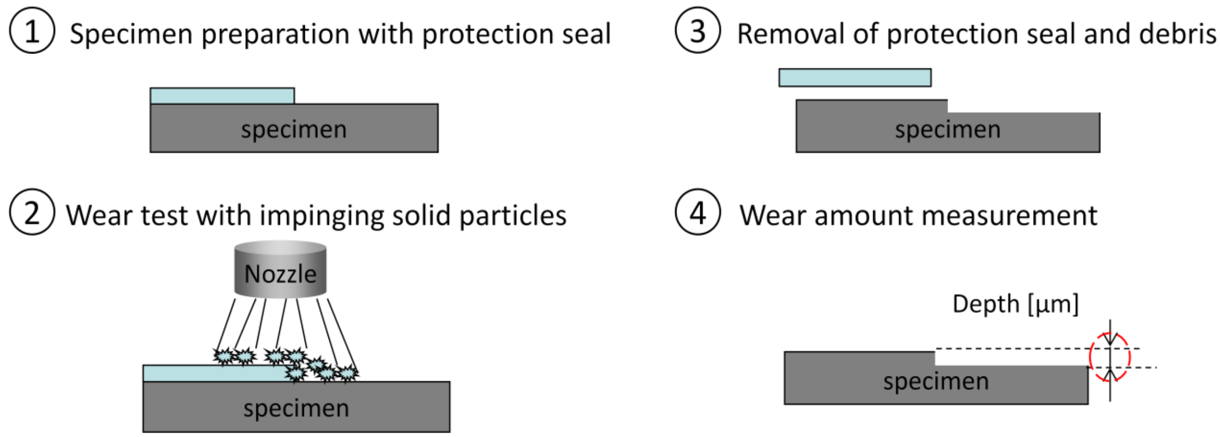

Figure 6.Procedure of wear amount measurement after each erosion step

3. RESULTS AND DISCUSSION

3.1. Erosion performance and material analysis

Erosive wornsurface of various SiC/SiC composite specimensare observed by direct-eye as illustrated in figure 7. The pores in CVI material, and traces of reinforced-fibre in newly fabricated composites are conspicuously stand out compared to commercial NITE composite. Erosion wear result shows that newly fabricated SiC/SiC composite has reasonable wear resistance compared to the two commercial composites (see figure 8). Erosion performance of newly fabricated composites,especially for the final product (LOT#17) was superior almost twice more resistance against commercial product by CVI (chemical vapour infiltration) route. Cef-NITE^{TM}fibre-reinforced NITE composite shows the best erosion resistance whilethe final product (LOT#17) shows only 20 % inferior to Cera-NITE^{TM} performance.

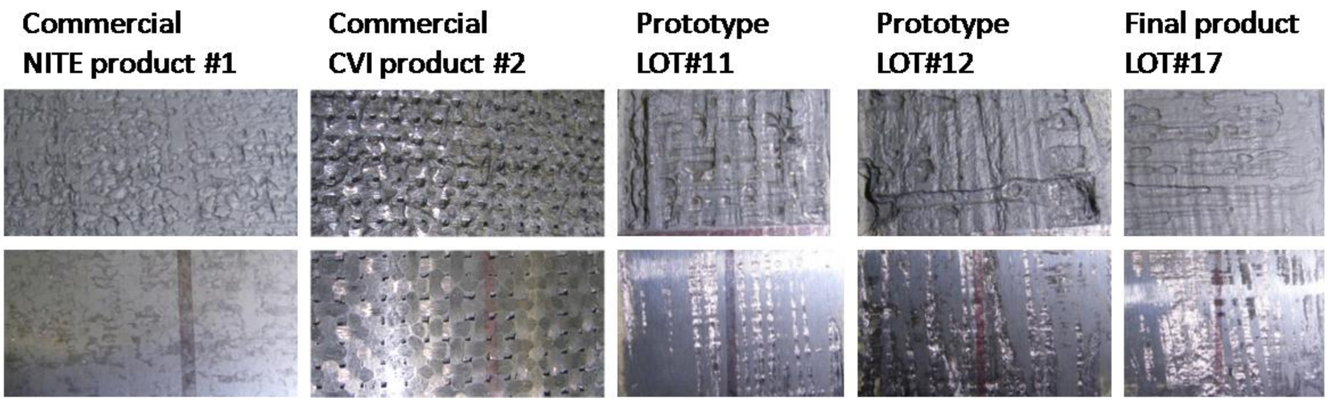

Figure 7. Bare eye observation on the worn surfaces of composites before and after the erosion test.

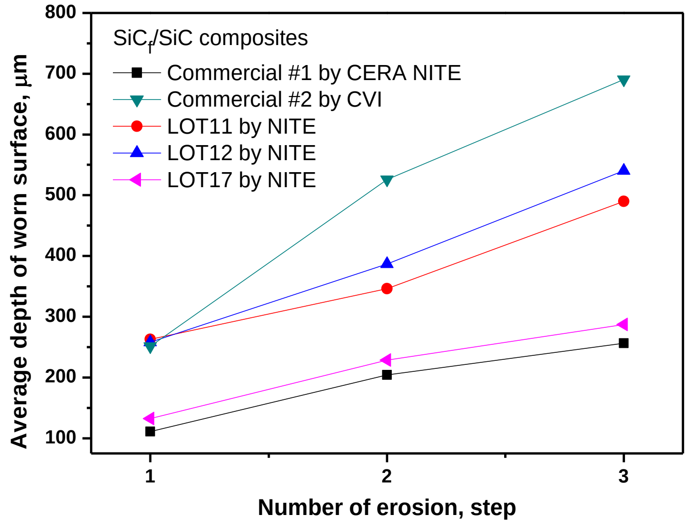

Figure 8. Wear rate under solid particle erosion test for various SiC/SiC composites.

Prototype material LOT#11 has a highervolume fraction about treble thePyC compared to the final product LOT#17,figure 9shows the amount of PyC presence between the fibre tows. SE micrograph in back scattered electron imaging (BEI) mode indicates fibres and PyC in a colour of grey and black, respectively. Severe fibre deformation and micro-pore formationare observed in prototype LOT#11.Increase of PyC amount results in severe fibre deformation and/or pore generation, which plays an important role for the erosive wear performance. Despite the PyC amount, LOT#12 shows the worst erosion performance. It is because of the fabrication methods and material properties, the related parts are described in the author's other papers [67-71]. In short, it results from the porosity as shown in figure 10.

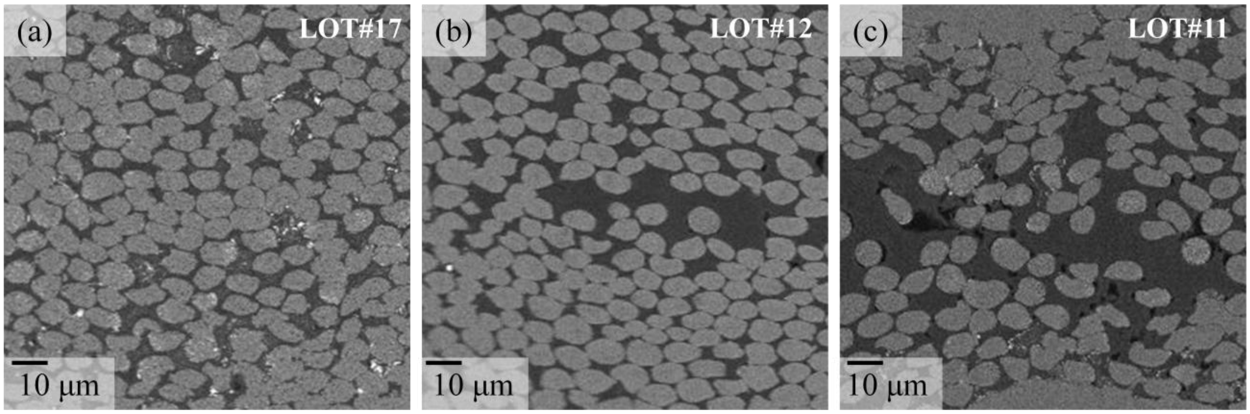

Figure 9.Employed amount of PyC, in (a) Final product, and in prototype materials (b), and (c).

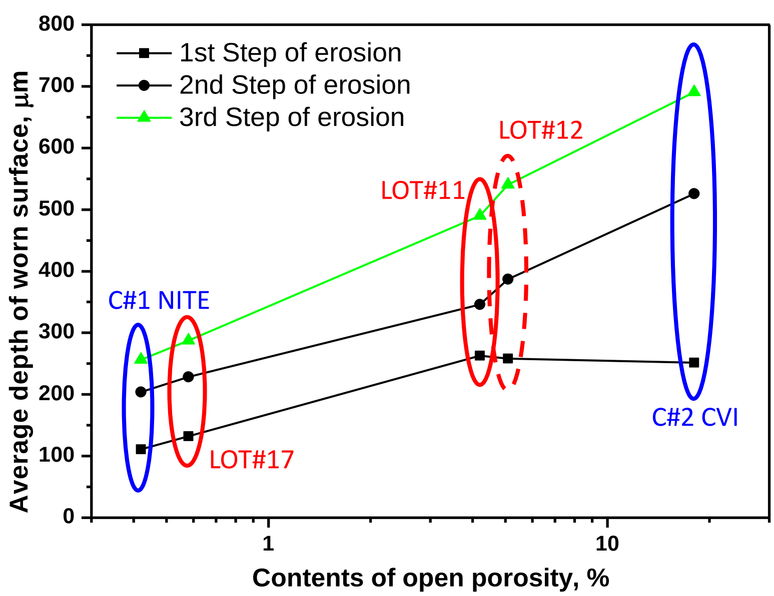

Figure 10Wear rate as a function of open porosity.

Figure 11 shows conspicuous defects in prototype material; a) irregular fibre deformations, b) insufficient densification of the matrix, c) distribution of residual oxide, d) micro-pores, e) deformation and crack formation on PyC, f) weak bonding strength between fibre and interface, and g) melted-flowed oxide phase around the fibre.

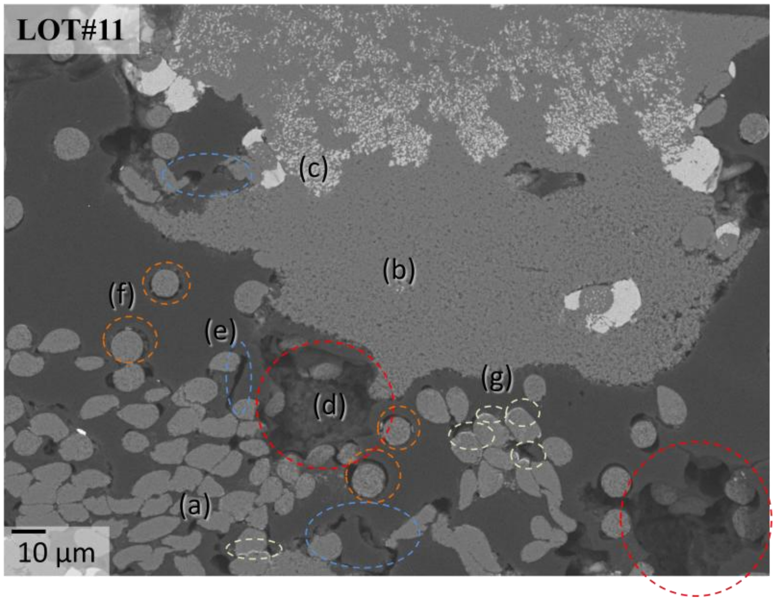

Figure 11. Conspicuous defects observed in typical region of prototype specimen LOT#11.

3.2. Wear behaviours and wear mechanisms

Two dominant erosive wear mechanisms were observed in this study, one is delamination of constituents.Another isfragmentation and detachment of constituents, the former usually caused by erosive crack propagation and later caused by an erosive impact.

A unit size of delamination is determined by the crack propagation length,mostly along the interface of constituents.It is the mostdecisive factor onthe amount ofwear. When a crack reaches to the interfacetwo kinds of crack branching mechanism will simultaneously occurs. In case of the strong bonded interface, the crack neitherpropagates to the next crack nor results in any delamination. However, in the opposite case,the crack easily propagates to the next crackand consequently,causinghuge delamination. It is observed that most of the erosive crack propagates through the weakest points.Evidential proof was confirmed in two prototype materialsthat the fragmentation of matrix and detachment of fibre were conspicuously observed in plenty of areas, which results from weak bonding strength. The final product was relatively strong in bonding strength, so that the fragmentation and detachment of those matrix and fibre were hardly observed. Comparatively, fragmentation of matrix and fibrewas rarely occurred by an erosive impact. In case of strong bonding between the fibre and interface the material can absorb the impact energy below critical and the critical energy depends on the bonding without crack propagation along the interface or delamination of the fibre. This leads to gradual wear of the constituents, as shown in figure 14b. In the final product LOT#17 the fragmentation of constituents is the dominant factor, due to the strong bonding among all constituents.

3.2.1. Wear on matrix

Different erosive wear behaviour was observed on new composites, as depicted in figure 12. Eroded surface of new composites by SEM observation shows the great difference between the coarse matrix and dense matrix. Due to inadequately applied pressure, phenomena of insufficient densification of matrix and scarce grain growth were observed in prototype LOT#11. Dense matrix also can be found in prototype materials; however the size of grain shows that their growth was relatively premature. Grain size of final product was relatively larger according to sufficient grain growth. Both grain pull-out and intergranular fractures by erosive impact were observed in eroded surface of final product (see figure 12d).

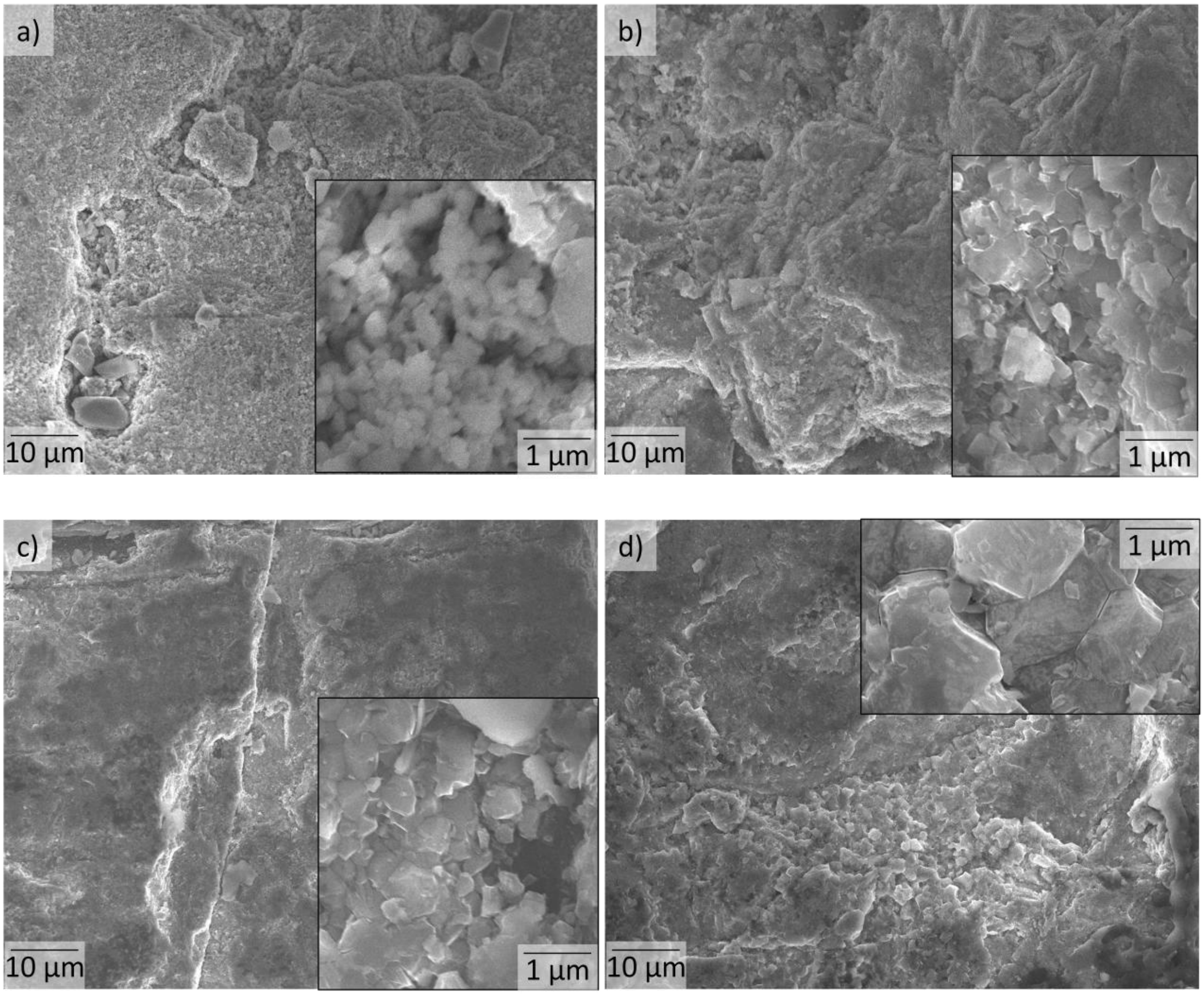

Figure 12.SE micrographs of eroded surface on (a) coarse matrix of prototype LOT#11, and a typical eroded region of (b) prototype LOT#11,(c) LOT#12, and (d) final product LOT#17.

Microscopic observation on the cross section of prototype materials also confirmed the porosities in the matrix. As shown in figure 13, (b) showspores between remnant agglomerates compared with (a) relative small amount of pores in the dense matrix. These inadequately pressurized areas and insufficient grain growth are one of the reasons of severe matrix fragmentation.

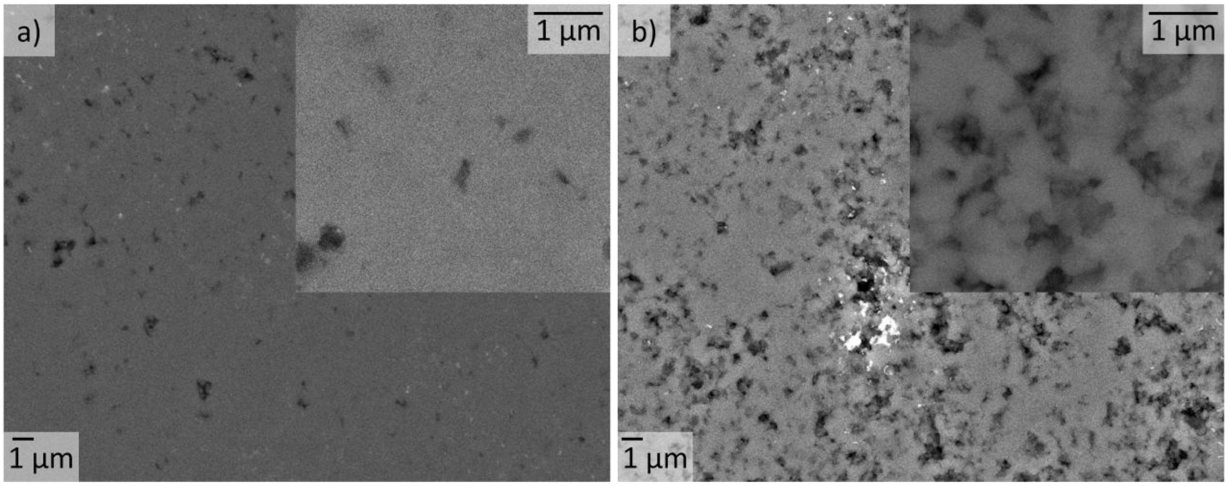

Figure 13. SE micrographs (BEI mode) of typical porosities inprototype LOT#11

(a) pores in dense matrix and (b) coarse matrix.

3.2.2. Wear on fibreand interface

Detachment of fibre was conspicuously observed in prototype materials. As a form of aperture between fibre and PyC which result in fibre separation, porosity caused easy fibre detachment even by a single impact. It was mainly generated by volume contraction around 24% of Pre-SiC fibre itself during the crystallization process. The effect of fibredetachment caused by weak bonding can be seen in figure 14a, where exact shaped and sized traces of fibre detachment can be found as the evidence. PyC cleavage was also observed, which was generated while the hot-pressing process.

For final product LOT#17, bonding strength between fibre and interface was relatively strong thatmost of the wear was result from gradual eroding (see figure 14b). Consequently, behaviours such as sudden detachment of constituents were rarely observed. These porosities influenced on a bonding strength of each constituent, which results in severe detachment of fibre and fragmentation of interface by an erosive impact (also shown in figure 16).

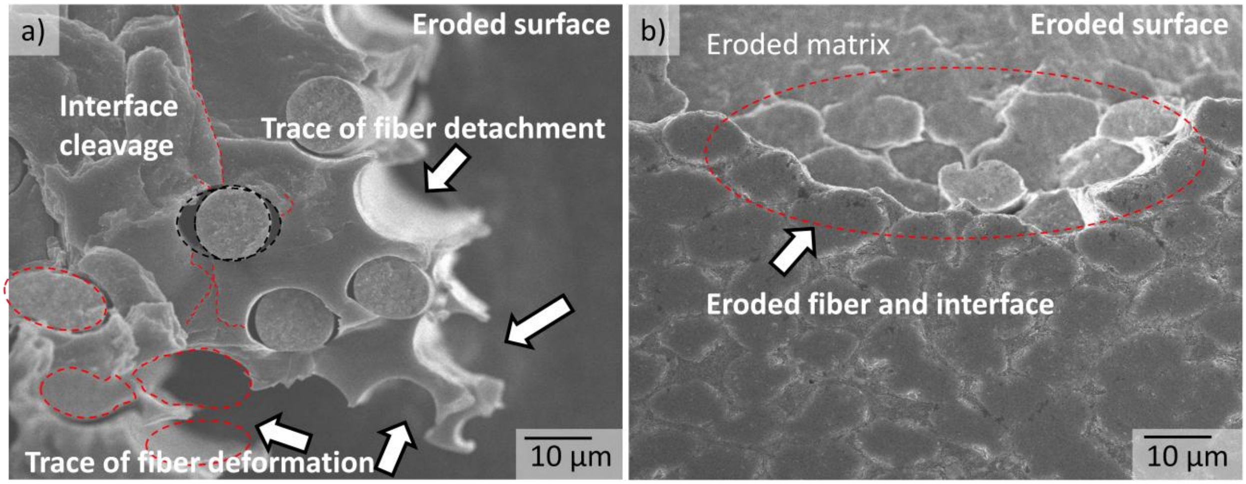

Figure 14.SE micrographs of fibre detachment and deformation observed in (a) prototype LOT#11, and (b) final product LOT#17

3.3. Erosion wear mechanism models for new SiC/SiC composites

Unit length of erosive crack propagation and confluence of cracks dominate the unit size of constituent delamination. Figure 15 shows the sequence of minor and huge erosion including delamination of each constituent.

Huge erosion occurs when propagation of erosive crack reaches to the interlayer of constituents where the bonding interface is fragile, and after that the crack branches along the matrix-interface. Erosive impact on α site sequentially leads to an erosion of matrix, a propagation of crack, and a huge unit of delamination, illustrated as α1, α2, and α3, respectively.

Minor erosion occurs when the erosive crack propagates and joined to next confluent crack, marked as β and γ. Erosive crack propagate further and jointo other cracks are also often occurred in the coarse matrix, marked as β.Sequential eroding on γ site shows a propagation of crack on the PyC, a crack propagation to aperture or weak bonding between fibre and PyC, and a crack connection to PyC cleavages, illustrated as γ1, γ2, and γ3, respectively.

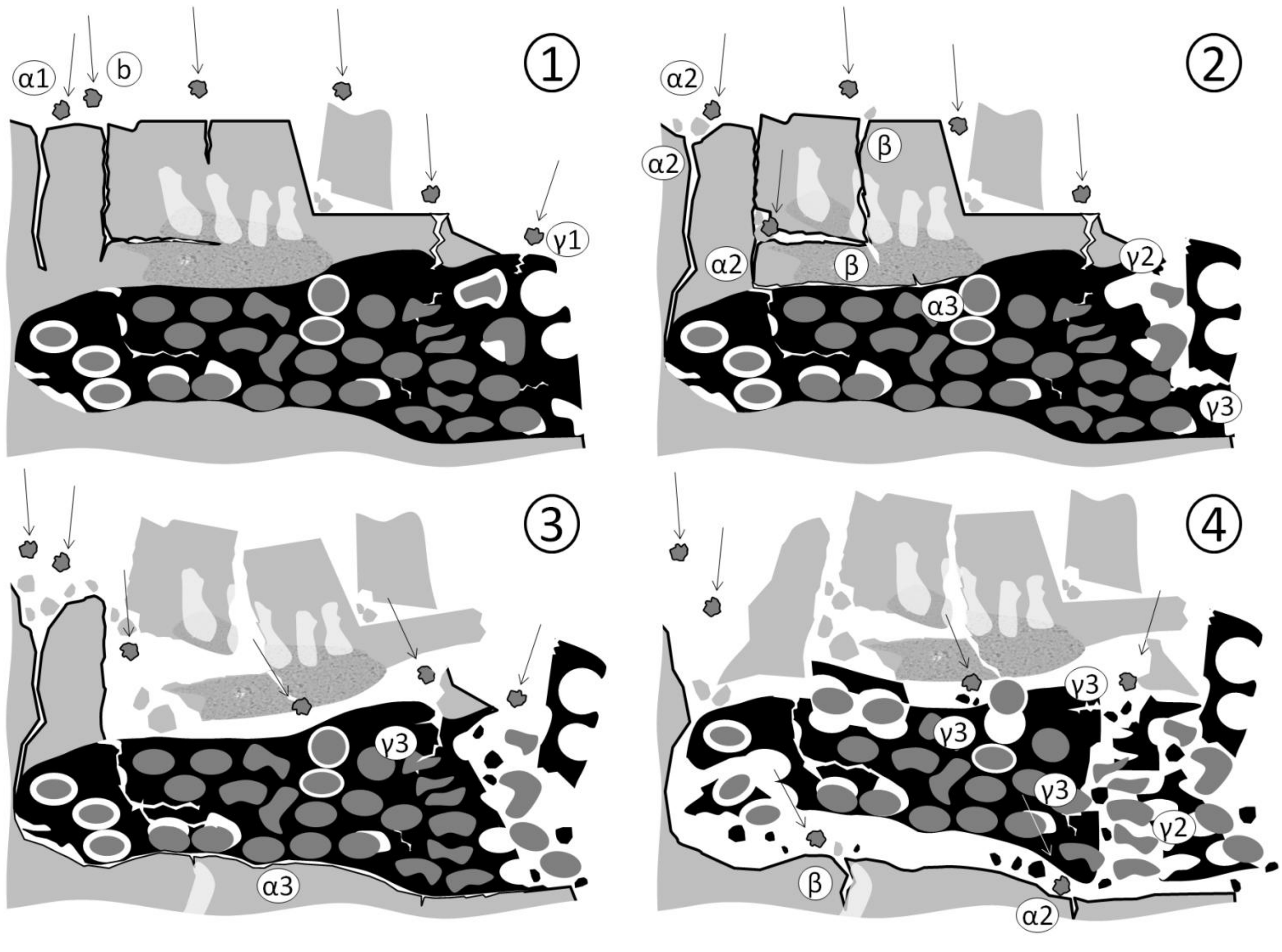

Figure 15.Cross sectional view of the erosive wear on the prototype of a new SiC/SiC composite. Sequential wear of each constituent by erosive crack propagation is shown in this model.

Erosion sites α, β, and γ are shown in micrograph figure 16 for the prototype LOT#11. In α site, the huge impact energy already sweeps through, so that only the eroded matrix and some remains of constituent parts of PyC and fibre are shown.Due to the defects in matrix, resultant behaviourare shown in β site as the matrix eroding and fragmentation, which will soon uncover the underneath constituents. In γ site, plenty of fibre detachments were observed with eroding of PyC.

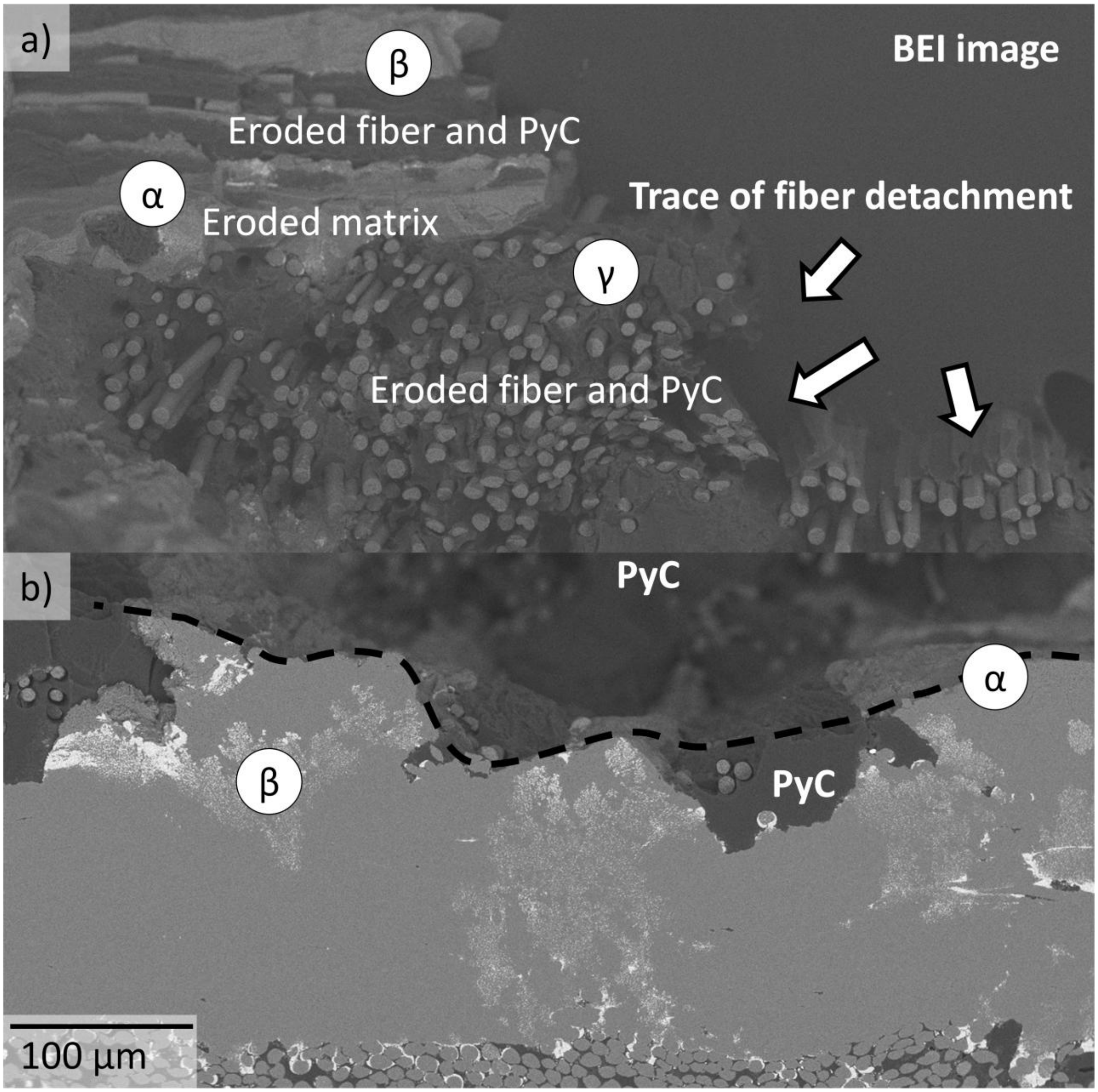

Figure 16. Erosive wear on each constituent observed in prototype LOT#11 a) focused on eroded fibres and PyC, and b) another focus on eroded matrix where huge delamination occurred.

Figure 17 shows the dominant erosive wear mechanisms on various SiC/SiC composites. Each number represents following erosive behaviours: 1. Matrix eroding and crack initiation, 2. Large and small unit of matrix fragmentation, 3. Interface and/or fibre eroding and cracking, 3a. PyC cleavage by eroding, 4.Fibre detachment, 5. Crack propagation along the interface and/or through the matrix, 5a. Through a weak interface between matrix and PyC layer, and 5b.Through a weak coarse matrix.

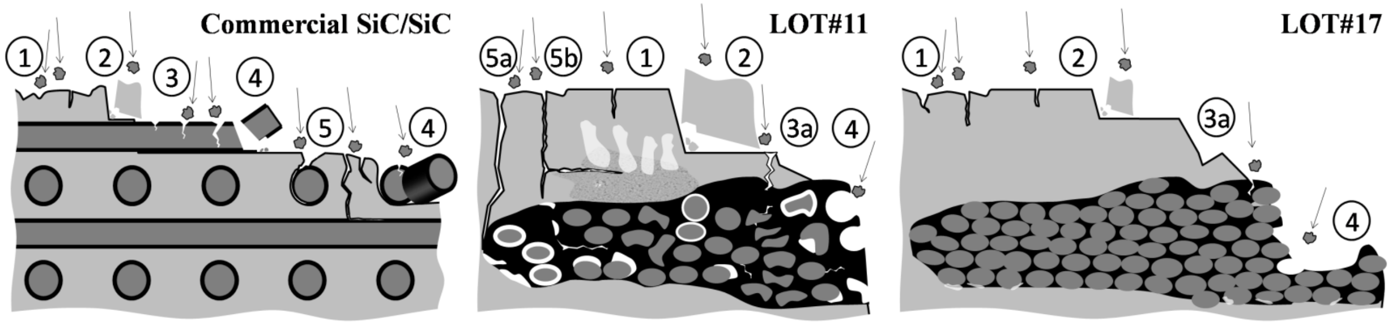

Figure 17 Cross sectional view of the erosive wear on SiC/SiC composites

4. CONCLUSION

Erosive wear test was carried out on a number of SiC/SiC composites. Schematic wear models are proposed with following details. Two dominant erosive wear mechanisms were observed for newlyfabricated SiC/SiC composites, one is delamination of constituents, which mainly occurred by crack propagation through the weakest interlayer. Another is fragmentation and detachment of constituents by an erosive impact. A unit size of delamination dominated by the erosive crack propagation length, which mostly determine the amount of wear volume.

The fabrication improvements have been successfully made for a newly fabricated SiC/SiCcomposite, as the result, the known defects are rarely observed in final product. Crucial fabrication factors for erosion wear resistance were; 1. Weak bonding strength of constituent parts, 2.Poor densification of matrix and scarce grain growth, 3.Interface cleavage with micro-pores, and4. Aperture along the fibre-interface

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